

Bureau of Mines Report of Investigations/1987

# Recovery of Phosphate From Florida Phosphate Slimes

By B. E. Davis, E. G. Davis, and T. O. Llewellyn





# Recovery of Phosphate From Florida Phosphate Slimes

By B. E. Davis, E. G. Davis, and T. O. Llewellyn

UNITED STATES DEPARTMENT OF THE INTERIOR Donald Paul Hodel, Secretary

BUREAU OF MINES
David S. Brown, Acting Director

Library of Congress Cataloging in Publication Data:

## Davis, B. E. (Broderick E.)

Recovery of phosphate from Florida phosphate slimes.

(Report of investigations; 9110)

Bibliography: p. 13.

Supt. of Docs. no.: I 28.23: 9110.

1. Phosphates—Florida. 2. Flotation. I. Davis, E. G. (Edward George), 1932- . II. Llewellyn, T. O. III. Title. IV. Series: Report of investigations (United States. Bureau of Mines); 9110.

TN23.U43 [TN914.U6F6] 622 s [661'.43] 87-600075

## CONTENTS

		Page
Intr Samp Hydre Flot Sam Sam Sam Sam Sam	ract	1 2 2 3 8 8 10 11 11 12 12
	TI I HOMBA TITONO	
	ILLUSTRATIONS	
1. 2. 3. 4.	Particle size distribution of sample 1	4 5 7 9
1. 2. 3.	Chemical analysis of head samples	3 4 5
4. 5.	Hydrocyclone test results for sample 1	6 7
	Hydrocyclone test results for sample 2	8
	Hydrocyclone underflow size analysis for sample 2	9
8.	Flotation results for sample 1, plus 37-µm flotation feed	10
	Flotation results for sample 1, plus 20-µm flotation feed	10
	Locked-cycle test series results for sample 1, plus 20-µm flotation feed.	10
	Flotation results for sample 2, plus 37-µm flotation feed	11
	Flotation results for sample 2, plus 20-µm flotation feed	11
. J.	imental phosphate collector	12

### UNIT OF MEASURE ABBREVIATIONS USED IN THIS REPORT

gal gallon lb/st pound per short ton

gal/min gallon per minute µm micrometer

in inch mt metric ton

1b pound pct percent

 $1bf/in^2$  pound force per square inch wt pct weight percent

### RECOVERY OF PHOSPHATE FROM FLORIDA PHOSPHATE SLIMES

By B. E. Davis,1 E. G. Davis,2 and T. O. Llewellyn2

#### ABSTRACT

The Bureau of Mines conducted tests on samples of two Florida phosphate slimes to devise a technique to recover phosphate currently discarded because of its fine size. A flotation technique was devised to recover the phosphate contained in two fractions of each slime: the plus 37- $\mu$ m fraction and the plus 20- $\mu$ m fraction. The technique involved conditioning the flotation feed with sodium silicate and a fatty acid-fuel oil emulsion, floating a rougher phosphate concentrate, and cleaning the concentrate in two cleaner stages with sodium silicate added to each for gangue depression. Concentrates containing 21.0 to 32.3 pct  $P_2O_5$  were produced with recoveries of 68.1 to 84.2 pct of the  $P_2O_5$  contained in the flotation feed.

<sup>1</sup>Minerals engineer.

<sup>&</sup>lt;sup>2</sup>Metallurgist.

Tuscaloosa Research Center, Bureau of Mines, Tuscaloosa, AL.

#### INTRODUCTION

One goal of the Bureau of Mines is to develop technology to enhance the conservation of domestic resources through more complete recovery of minerals. In an effort to conserve domestic mineral resources through increased utilization of process wastes, the Bureau has conducted research to establish mineral beneficiation technology for recovering phosphate from phosphate slimes. Of the 53.6 million mt of phosphate rock produced in the United States in 1981, 86.3 pct was produced in Florida and North Carolina (1).3

Currently phosphate is mined from the Bone Valley Formation in central Florida. Overburden is stripped with a dragline until the phosphate ore, known as matrix, is reached. The matrix is mined with the dragline and dumped into a slurry pit. The matrix is slurried with high-pressure hydraulic jets and pumped via a pipeline to the beneficiation plant. During beneficiation the minus 105-µm material is removed from the matrix by hydrocyclones. A typical matrix contains about 27 wt pct minus 105-µm material, principally clay minerals (2). The aqueous suspension of these fines, which is called slimes, is discharged from the washer plant at 2 to 6 pct solids. The size range of these particles is 0.01 to 105 µm, with the median size being about 1 µm. Phosphate slimes contain 6 to 17 pct P2O5, which in

some cases amounts to almost one-third of the phosphate in the matrix (3). Recovery of phosphate from the slimes would lengthen the life of domestic phosphate reserves.

Florida phosphate slimes have been extensively characterized by the Bureau of Mines and found to be variable with respect to their solids content, size, and clay content, and the rate at which they dewater (4). The Bureau also conducted tests on seven samples of Florida phosphate slimes to develop a method to recover the phosphate contained in the plus 37-µm fraction of the waste product (5). The method consisted of a system of hydrocyclones and hydroseparators to recover the plus 37-µm material, which was subsequently subjected to fatty acid flotation.

Over the years many attempts have been made to define and to explain what flotation operators call "the slimes problem" ( $\underline{6}$ ). Much progress has been made in the past few years in fine-particle flotation, yet few applications have occurred in the mineral industry and none in the phosphate industry. As a result, the Bureau conducted research to recover phosphate contained in the plus  $37-\mu m$  and plus  $20-\mu m$  fraction of two samples of phosphate slime. This report summarizes the results of the investigation.

#### SAMPLES DESCRIPTION

Two large samples of phosphate slime were collected from operations in Florida. Site selection was based on preliminary characterization of waste from five mines. Sample 1 was collected from a slime waste impoundment that had partly dewatered. The sample was collected

using a submersible pump to obtain a vertical cross section of the pond. Approximately 1,300 gal of sample 1 containing 10 to 12 pct solids was pumped into 55-gal drums and transported to Tuscaloosa. Sample 2 was collected at a beneficiation plant. The sample was collected directly from the primary hydrocyclone overflow and contained approximately 3.5 pct solids. About 1,450 gal of sample 2 was collected for research studies.

<sup>&</sup>lt;sup>3</sup>Underlined numbers in parentheses refer to items in the list of references at the end of this report.

X-ray diffraction analysis of sample 1 showed that it consisted mostly of apatite, quartz, and smectite with minor amounts of dolomite and kaolinite. Trace amounts of wavellite, crandallite, and illite were also detected. Sample 2 contained essentially the same minerals as sample 1 except that no wavellite was detected. Table 1 presents the chemical analysis of the samples.

One important characteristic of the phosphate slime is the particle size distribution, which was determined by settling based on Stoke's law and also with the L&N MICROTRAC4 particle size analyzer. Comparable results were obtained with both methods. As expected, both samples contained significant amounts of material in the low-micrometer size range. Sample 1 contained 39 pct minus  $5-\mu m$  material accompanied by 39.9 pct of the  $P_2O_5$  in the sample. Table 2

gives detailed results of the size analysis of sample 1, and figure 1 is a graph of its particle size distribution. Sample 2 was finer than sample 1 and contained 78.2 pct minus 5- $\mu$ m material. A significant amount of the  $P_2O_5$ , 66.0 pct, reported to this size fraction of the sample. Table 3 presents the size analysis of sample 2, while figure 2 is a graph of its particle size distribution.

TABLE 1. - Chemical analysis of head samples, percent

	Sample 1	Sample 2
P <sub>2</sub> O <sub>5</sub>	13.0	5.9
Ca0	13.3	9.7
MgO	.8	1.6
CO <sub>2</sub>	8.6	2.2
Si0 <sub>2</sub>	33.6	42.1
$A1_2\bar{0}_3$	13.5	12.0
Fe <sub>2</sub> 0 <sub>3</sub>	.9	3.8

#### HYDROCYCLONE CLASSIFICATION

The objective of the flotation studies was to recover the phosphate contained in the plus  $37-\mu m$  and plus  $20-\mu m$  fractions of each slime. The minus  $20-\mu m$  material had to initially be removed, and then the minus  $37-\mu m$  material had to be removed

for flotation studies of the plus  $37-\mu m$  fraction. A two-stage continuous hydrocyclone classification circuit was used to remove a significant amount of the undersize material. Plus  $37-\mu m$  and plus  $20-\mu m$  size separations were completed in the laboratory by screening and sedimentation, respectively, for flotation feed samples.

<sup>&</sup>lt;sup>4</sup>Reference to specific products does not imply endorsement by the Bureau of Mines.

TABLE 2. - Size analysis of sample 1

		7	1 - <del>-</del> -		T 66	T 010	1110
Size fraction, µm	wt pct	P <sub>2</sub> O <sub>5</sub>	Ca0	Mg0	CO <sub>2</sub>	SiO <sub>2</sub>	A1 <sub>2</sub> 0 <sub>3</sub>
		C	hemical	analysi	s, pct		
Plus 105	3.8	3.3	5.1	0.5	1.9	84.4	1.1
Minus 105 plus 75	4.7	6.9	8.9	.5	1.9	69.8	2.2
Minus 75 plus 53	3.5	8.0	12.4	.5	3.8	64.2	2.3
Minus 53 plus 37	5.0	9.9	17.6	1.3	6.6	49.5	3.0
Minus 37 plus 20	10.0	12.6	25.9	2.5	12.7	33.1	3.5
Minus 20 plus 10	15.0	18.2	26.0	1.3	11.2	23.1	5.7
Minus 10 plus 5	19.0	13.3	7.9	.2	7.7	14.3	21.1
Minus 5	39.0	13.2	8.8	•5	9.2	30.1	20.4
Composite	100.0	12.9	13.3	.8	8.6	32.4	13.5
			Distri	bution,	pct		***************************************
Plus 105	3.8	1.0	1.5	2.3	0.8	9.9	0.3
Minus 105 plus 75	4.7	2.5	3.1	2.9	1.0	10.1	.8
Minus 75 plus 53	3.5	2.2	3.3	2.3	1.5	6.9	.6
Minus 53 plus 37	5.0	3.8	6.6	8.0	3.8	7.6	1.1
Minus 37 plus 20	10.0	9.8	19.4	30.8	14.7	10.2	2.6
Minus 20 plus 10	15.0	21.2	29.2	24.0	19.5	10.7	6.3
Minus 10 plus 5	19.0	19.6	11.2	4.7	17.0	8.4	29.6
Minus 5	39.0	39.9	25.7	25.0	41.7	36.2	58.7
Total	100.0	100.0	100.0	100.0	100.0	100.0	100.0

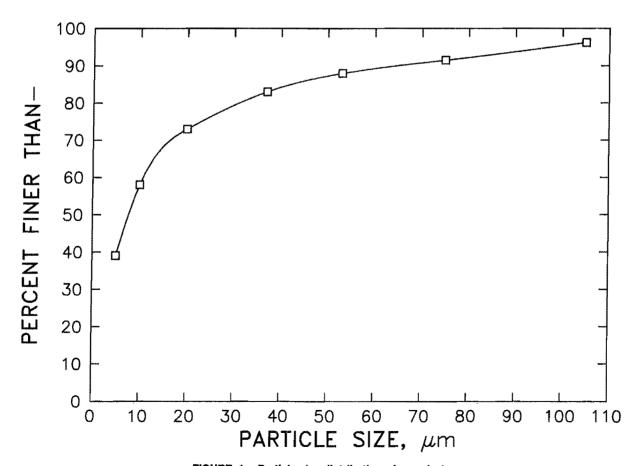


FIGURE 1.—Particle size distribution of sample 1.

TABLE 3. - Size analysis of sample 2

Size fraction, µm	wt pct	P <sub>2</sub> O <sub>5</sub>	CaO	MgO	CO <sub>2</sub>	SiO <sub>2</sub>	A1 20 3	Fe <sub>2</sub> O <sub>3</sub>
			Chem	cal ana	lysis,	pct		<del></del>
Plus 105	3.4	17.4	30.4	0.9	4.1	23.4	3.2	1,.3
Minus 105 plus 37	7.1	9.2	14.4	.9	1.8	44.9	6.8	2.1
Minus 37 plus 20	3.2	9.4	15.9	1.2	1.4	42.9	6.4	2.3
Minus 20 plus 10	4.2	7.2	12.8	1.4	1.7	41.7	9.8	2.5
Minus 10 plus 5	3.9	6.3	10.4	1.5	1.8	44.9	11.6	2.7
Minus 5	78.2	5.2	8.8	1.5	1.2	43.8	13.9	2.8
Composite	100.0	6.2	10.4	1.4	1.4	43.1	12.5	2.7
	,		Dis	stributi	on, pct			,
Plus 105	3.4	9.5	9.9	2.1	9.9	1.8	0.9	1.6
Minus 105 plus 37	7.1	10.7	9.9	4.5	9.2	7.4	3.9	5.6
Minus 37 plus 20	3.2	4.9	4.9	2.7	3.2	3.2	1.6	2.8
Minus 20 plus 10	4.2	4.9	5.2	4.1	5.1	4.1	3.3	4.0
Minus 10 plus 5	3.9	4.0	3.9	4.1	5.1	4.1	3.6	4.0
Minus 5	78.2	66.0	66.2	82.5	67.5	79.4	86.7	82.0
Total	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

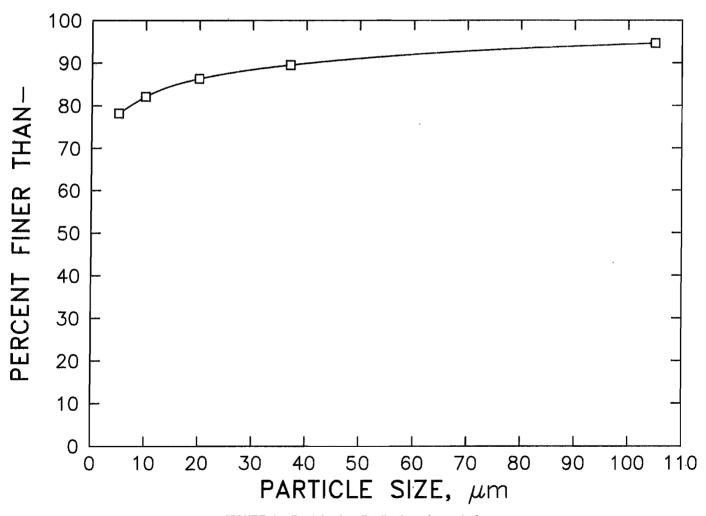


FIGURE 2.—Particle size distribution of sample 2.

TABLE 4. - Hydrocyclone test results for sample 1

Product	wt pct	Chemical analysis, pct		Distribution, pct	
		P <sub>2</sub> O <sub>5</sub>	S10 <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	SiO <sub>2</sub>
Overf1ow	65.3	12.0	33.7	68.9	54.6
Underflow	34.7	10.2	52.7	31.1	45.4
Composite	100.0	11.5	39.3	100.0	100.0

Sample 1 was diluted to 3 pct solids for hydrocyclone classification. A cluster of four 1-in-diam hydrocyclones was used in the first stage for sample 1. The hydrocyclones were operated at 50 1bf/in<sup>2</sup> with a feed rate of approximately 16 gal/min of 3-pct-solids slime. The first stage cluster of hydrocyclones produced an underflow-overflow ratio of 45:55 (dry solids basis). The underflow was fed to the second-stage single 1-indiam hydrocyclone, which was operated at 20 lbf/in2. Approximately 65 wt pct (dry solids) of the material fed to the second-stage cyclone reported to the underflow. The overflow was recycled to the feed for the first-stage hydrocyclones. Table 4 summarizes the hydrocyclone classification and shows that 65.3 wt pct (dry solids) of the material was rejected with a loss of 68.9 pct of the P<sub>2</sub>O<sub>5</sub>. However, the minus 5-µm content

of the slime was reduced from 39 to 16 pct. Table 5 gives the size analysis of the hydrocyclone underflow for sample 1, and figure 3 is a graph of its particle size distribution.

Sample 2 was classified using a Mozely hydrocyclone test unit. The 2-in-diam hydrocyclone was made of polyurethane. It was operated at 50 1bf/in2 with a feed rate of approximately 8.2 gal/min of 3.5 pct solids. The underflow was subjected to a second-stage classification under the same conditions as the first stage. The classification process rejected 91.8 wt pct (dry solids) of the material with a P2O5 loss of 86.4 pct. Table 6 summarizes the results of the hydrocyclone classification process for sample 2. Table 7 shows that the process reduced the minus 5-um content of the slime from 78.2 to 23.0 pct, and figure 4 is a graph of its particle size distribution.

TABLE 5. - Hydrocyclone underflow size analysis for sample 1

Size fraction, µm	wt pct	Chemical an	alysis, pct	Distribution, pc	
		P <sub>2</sub> O <sub>5</sub>	SiO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	SiO <sub>2</sub>
Plus 105	10	3.3	84.4	2.9	20.4
Minus 105 plus 53	18	7.4	67.4	11.6	29.3
Minus 53 plus 37	10	9.9	49.5	8.6	11.9
Minus 37 plus 20	17	12.6	33.1	18.6	13.6
Minus 20 plus 10	15	18.2	23.1	23.7	8.4
Minus 10 plus 5	14	13.3	14.3	16.2	4.8
Minus 5	16	13.2	30.1	18.4	11.6
Composite	100	11.5	41.4	100.0	100.0

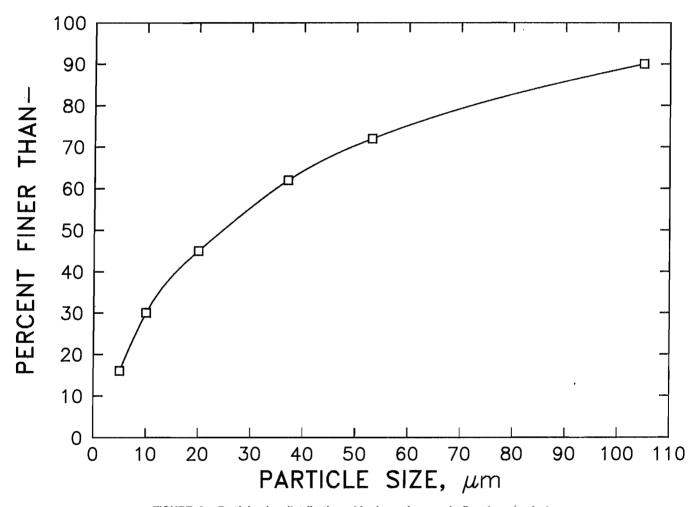


FIGURE 3.—Particle size distribution of hydrocyclone underflow for sample 1.

TABLE 6. - Hydrocyclone test results for sample 2

Product	Wt pct	Chemical an	alysis, pct	Distribution, pct	
		P <sub>2</sub> O <sub>5</sub>	SiO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	SiO <sub>2</sub>
Overflow	91.8	5.2	43.4	86.4	92.2
Underflow	8.2	9.2	41.3	13.6	7.8
Composite	100.0	5.5	43.2	100.0	100.0

#### FLOTATION

A general flotation technique was devised to recover the phosphate contained in the waste slimes. After sizing, flotation feed was conditioned with sodium silicate followed by conditioning with an anionic phosphate collector; conditioning times were varied to determine optimum times for each sample and feed size. After conditioning, a rougher phosphate concentrate was floated for approximately 3 min in a Denver laboratory flotation machine. The pH of the rougher flotation was adjusted using Na<sub>2</sub>CO<sub>3</sub> prior to the introduction of air to the flota-The rougher phosphate flotion machine. tation concentrate was cleaned and recleaned, with sodium silicate added to each cleaner for gangue depression. flotation products were dried, weighed, and analyzed.

Optimum flotation conditions were determined for each sample and each size of flotation feed. The sodium silicate dosage was varied from 0.1 to 5.0 1b sodium silicate per short ton of flotation feed in the conditioner and in the cleaner flotation stages. 5 The method of conditioning was also tested and included conditioning in the flotation machine, in a high-shear conditioner, and in a slowspeed conditioner. These represented medium-, high-, and low-shear conditions, respectively. Several different anionic phosphate collectors produced by Arizona Chemical, Emery, and Alcolac were tested. These collectors were added as pure reagents and as emulsions. Phosphate

collector dosage was varied to determine the optimum reagent dosage. The optimum conditions are reported for each sample and size of flotation feed. Industry requirements for acceptable concentrate are a ratio of (Al $_2$ O $_3$ +Fe $_2$ O $_3$ +MgO) to P $_2$ O $_5$ , all in weight percent, of no more than 0.1 and a minimum P $_2$ O $_5$  content of 28 pct (7). However, blending may still allow use of concentrates that do not quite satisfy these reqirements.

# SAMPLE 1, PLUS 37-µm FLOTATION FEED RESULTS

A flotation feed was conditioned at approximately 60 pct solids in a slow-speed conditioner for 2 min with 0.15 1b sodium silicate per short ton of flotation feed. The material was subsequently conditioned with 5.0 lb/st fatty acid-fuel oil emulsion for 5 min. The emulsion contained 15 parts Acintol FA 1, 10 parts No. 5 fuel oil, 2.5 parts NaOH, and 0.5 part EC 111 (sodium cetyl sulfate) as an emulsion stabilizer. The conditioned feed was added to the flotation machine, and tap water was added to give a pulp density of 25 to 30 pct solids. A rounger concentrate was floated at a pH of 9.0 for approximately 3 min. The rougher concentrate was cleaned and recleaned with 0.5 lb/st sodium silicate added to each cleaner stage. The final concentrate contained 25.7 pct P205, 5.8 pct  $SiO_2$ , 6.3 pct  $Al_2O_3$ , 1.1 pct  $Fe_2O_3$ , and 0.9 pct MgO with a  $P_2O_5$  recovery of 77.4 pct of the  $P_2O_5$  in the plus 37- $\mu$ m material. Table 8 gives the detailed results of the test.

<sup>&</sup>lt;sup>5</sup>All reagent dosages are reported as pounds per short ton of flotation feed.

TABLE 7. - Hydrocyclone underflow size analysis for sample 2

Size fraction, µm	wt pct	Chemical an	Distribut	tion, pct	
		P <sub>2</sub> O <sub>5</sub>	SiO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	SiO <sub>2</sub>
Plus 105	16.0	20.2	29.8	32.8	11.4
Minus 105 plus 37	21.4	10.7	45.2	23.2	23.2
Minus 37 plus 20	8.2	9.7	46.1	8.1	9.1
Minus 20 plus 10	20.2	8.4	42.1	17.2	20.4
Minus 10 plus 5	11.2	6.2	46.0	7.0	12.3
Minus 5	23.0	5.0	42.4	11.7	23.6
Composite	100.0	9.9	41.6	100.0	100.0

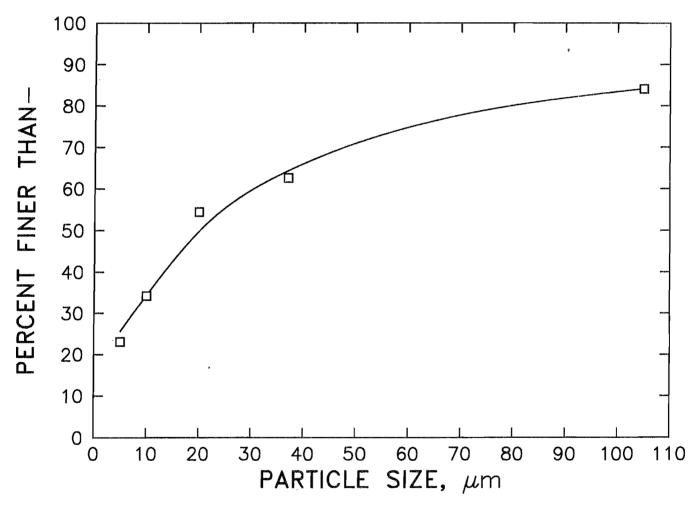


FIGURE 4.—Particle size distribution of hydrocyclone underflow for sample 2.

TABLE 8. - Flotation results for sample 1, plus 37-µm flotation feed

Product	wt pct	Chemical an	alysis, pct	Distribution, pct		
		P <sub>2</sub> O <sub>5</sub>	SiO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	SiO <sub>2</sub>	
Phosphate concentrate	21.2	25.7	5.8	77.4	1.8	
2d cleaner tailings	5.7	20.2	26.1	16.5	2.2	
lst cleaner tailings	3.9	3.8	75.3	2.1	4.4	
Rougher tailings	69.2	• 4	88.5	4.0	91.6	
Composite	100.0	7.0	66.9	100.0	100.0	

TABLE 9. - Flotation results for sample 1, plus 20-µm flotation feed

Product	wt pct	Chemical analysis, pct		Distribut	ion, pct
		P <sub>2</sub> O <sub>5</sub>	SiO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	SiO <sub>2</sub>
Phosphate concentrate	29.1	21.0	14.8	84.2	6.8
2d cleaner tailings	12.9	5.3	76.8	9.4	15.6
lst cleaner tailings	26.8	1.0	83.9	3.7	35.4
Rougher tailings	31.2	.6	85.8	2.7	42.2
Composite	100.0	7.3	63.5	100.0	100.0

TABLE 10. - Locked-cycle test series results for sample 1, plus 20-µm flotation feed

Product	wt pct	Chemical an	alysis, pct	Distribut	ion, pct
		P <sub>2</sub> O <sub>5</sub>	SiO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	SiO <sub>2</sub>
Phosphate concentrate	32.7	22.2	9.4	86.0	5.1
2d cleaner tailings	2.8	13.3	45.2	4.5	2.1
lst cleaner tailings	4.9	6.1	68.4	3.5	5.5
Rougher tailings	59.6	•9	88.1	6.0	87.3
Composite	100.0	8.4	60.2	100.0	100.0

# SAMPLE 1, PLUS 20-µm FLOTATION FEED RESULTS

A plus 20- $\mu$ m flotation feed was conditioned in a slow-speed conditioner with 0.15 lb/st sodium silicate for 2 min and with 8.0 lb/st fatty acid-fuel oil emulsion for 5 min at approximately 60 pct solids. The conditioned feed was added to the flotation machine, with tap water added to bring the froth level to the flotation cell overflow. A rougher phosphate concentrate was floated at a pH of 9.0. The rougher concentrate was cleaned and recleaned with 0.5 lb/st sodium silicate added to each cleaner for gangue depression. The final concentrate contained 21.0 pct  $P_2O_5$  and 14.8 pct SiO<sub>2</sub>

with an attendant recovery of 84.2 pct of the contained  $P_2O_5$  (table 9). A lockedcycle test series was conducted to simulate continuous flotation of the plus 20um material of sample 1 (table 10). The flotation conditions used were optimum conditions determined for the sample. The second cleaner tailings of each test were added to the flotation feed for the subsequent test. The series was concluded after seven tests. The concentrate produced in the final test contained 22.2 pct  $P_2O_5$  and 9.4 pct  $SiO_2$ . The  $P_2O_5$  recovery was 86.0 pct of the contained  $P_2O_5$ . The results indicate that continuous tests would produce comparable results to batch tests.

X-ray diffraction analysis of flotation concentrates from sample 1 revealed that wavellite and dolomite had been concentrated with the apatite. The presence of these minerals would make the concentrate questionable as an acidulation feed owing to the high amount of  $Al_2O_3$  and MgO. Chemical analysis of the concentrates revealed that they contained as high as 5.1 pct  $Al_2O_3$ , 1.2 pct  $Fe_2O_3$ , and 1.6 pct MgO.

# SAMPLE 2, PLUS 37-µm FLOTATION FEED RESULTS

A flotation feed sample of plus 37-µm material was conditioned with 0.5 lb/st sodium silicate in a slow-speed conditioner at approximately 60 pct solids for 2 min. Five pounds of fatty acid-fuel oil emulsion per short ton was added, and the pulp was conditioned for an additional 5 min. The conditioned feed was transferred to a flotation machine, and a rougher phosphate flotation was accomplished at pH 9.0. The rougher concentrate was cleaned and recleaned with 0.5 lb/st sodium silicate added to each

cleaner. The final concentrate contained 30.6 pct  $P_2O_5$ , 5.4 pct  $SiO_2$ , 1.8 pct  $Al_2O_3$ , 0.9 pct  $Fe_2O_3$ , and 0.2 pct MgO with an attendant recovery of 73.5 pct of the contained  $P_2O_5$  (table 11).

# SAMPLE 2, PLUS 20-µm FLOTATION FEED RESULTS

A flotation feed sample of plus 20-µm material was conditioned with 0.1 1b/st sodium silicate for 2 min. A slow-speed conditioner was used, and a pulp density of about 60 pct solids was maintained. Eight pounds of fatty acid-fuel oil emulsion per short ton was added, and the feed was conditioned for an additional 5 The conditioned feed was transferred to a flotation machine, rougher concentrate was floated at pH The rougher concentrate was cleaned and recleaned with 0.5 lb/st sodium silicate added to each cleaner for gangue depression. The final concentrate contained 29.4 pct  $P_2O_5$ , 6.8 pct  $SiO_2$ , 3.1 pct  $A1_2O_3$ , 1.2 pct  $Fe_2O_3$ , and 0.3 pct MgO with a recovery of 68.1 pct of the contained  $P_2O_5$  (table 12).

TABLE 11. - Flotation results for sample 2, plus 37-µm flotation feed

Product	Wt pct	Chemical an	alysis, pct	Distribution, pct		
		P <sub>2</sub> O <sub>5</sub>	SiO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	SiO <sub>2</sub>	
Phosphate concentrate	38.9	30.6	5.4	73.5	5.2	
2d cleaner tailings	10.8	24.6	15.5	16.5	4.2	
1st cleaner tailings	16.3	6.5	65.5	6.6	26.7	
Rougher tailings	34.0	1.6	75.1	3.4	63.9	
Composite	100.0	16.1	40.0	100.0	100.0	

TABLE 12. - Flotation results for sample 2, plus 20-µm flotation feed

Product	wt pct	Chemical an	alysis, pct	Distribution, pct	
		P <sub>2</sub> O <sub>5</sub>	S10 <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	S10 <sub>2</sub>
Phosphate concentrate	33.5	29.4	6.8	68.1	5.2
2d cleaner tailings	17.5	16.1	39.2	19.5	15.6
1st cleaner tailings	21.2	4.8	71.5	7.0	34.6
Rougher tailings	27.8	2.8	70.2	5.4	44.6
Composite	100.0	14.5	43.8	100.0	100.0

TABLE 13. - Flotation results for sample 2, plus 20-µm flotation feed, using an experimental phosphate collector

Product	wt pct	Chemical an	alysis, pct	Distribution, pct	
	_	P <sub>2</sub> O <sub>5</sub>	SiO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	SiO <sub>2</sub>
Phosphate concentrate	41.0	32.3	6.5	84.1	6.1
Cleaner tailings	9.6	12.1	45.2	7.4	10.0
Rougher tailings	49.4	2.7	73.8	8.5	83.9
Composite	100.0	15.7	43.5	100.0	100.0

# SAMPLE 2, EXPERIMENTAL PHOSPHATE COLLECTOR RESULTS

During the research work, many new excollectors were perimental phosphate tested, only one of which showed positive results. It was Alcolac DV 1447 and was listed only as an anionic surfactant. The collector was water soluble and was used as a 25-pct solution. A flotation feed sample of plus 20-µm sample 2 material was conditioned for 2 min with 0.1 lb/st sodium silicate in a slow-speed conditioner at approximately 60 pct sol-The feed was conditioned for 5 min with 5.4 lb/st anionic surfactant and 2.6 1b/st fuel oil. The conditioned feed was added to a flotation machine, and a

rougher phosphate flotation was accomplished. The rougher concentrate only had to be cleaned once with 0.5 lb/st sodium silicate added. The final concentrate contained 32.3 pct  $P_2O_5$  and 6.5 pct  $SiO_2$ . The  $P_2O_5$  recovery was 84.1 pct of the contained  $P_2O_5$  (table 13). The experimental collector test results were obtained toward the end of the research project, and further testing was not possible.

X-ray diffraction analysis of flotation concentrates from sample 2 revealed that there was very little wavellite or dolomite in the concentrates. Chemical analysis of the concentrates showed that they contained approximately 2.0 pct  ${\rm Al}_2{\rm O}_3$  and 0.25 pct MgO.

#### CONCLUSIONS

Two large phosphate slime samples were obtáined from central Florida phosphate producers. The samples were classified with hydrocyclones to remove a majority of the water and minus 5-µm material. Plus 37-µm and plus 20-µm flotation feed samples were prepared in the laboratory by screening and sedimentation. A flotation technique to recover the phosphate contained in the flotation feed was devised. The technique consisted of conditioning at a high percentage of solids in a slow-speed conditioner with sodium silicate and a fatty acid-fuel oil emulsion. A rougher phosphate flotation was followed by cleaner and recleaner flotations to produce a phosphate concentrate. Concentrates were produced from sample 1

that contained up to 25.7 pct P205 with an attendant recovery of 77.4 pct and an overall recovery of 16.2 pct of the P205 in the slime. However, the presence of wavellite and dolomite in the concentrate could render it unsuitable as acidulation Concentrates were produced from sample 2 that contained up to 30.6 pct  $P_2O_5$  with an attendant recovery of 73.5 pct and an overall recovery of 17.1 pct of the P<sub>2</sub>O<sub>5</sub> in the slime. No significant amount of wavellite or dolomite was found in the concentrates of sample 2. viability of recovering phosphate from any given phosphate slime would probably rest upon the wavellite and dolomite content of the slime.

A concentrate that contained 32.3 pct  $P_2O_5$  with an attendant recovery of 84.1 pct of the contained  $P_2O_5$  was produced from sample 2 plus  $20-\mu m$  material using an experimental anionic surfactant as the

phosphate collector. Only one cleaner stage was required. These results could warrant further studies of recovering phosphate from waste slimes.

#### REFERENCES

- 1. Stowasser, W. F. Phosphate. Bu-Mines Mineral Commodity Profile, 1983, 18 pp.
- 2. Lawver, J. E., R. E. Snow, Sr., and B. J. Clingan. A Computerized System for Using Response Surface Methodology To Evaluate Phosphate Flotation Variables. Min. Eng., 1979, pp. 1230-1235.
- 3. U.S. Bureau of Mines. The Florida Phosphate Slimes Problem—A Review and a Bibliography. BuMines IC 8668, 1975, 41 pp.
- 4. Lamont, W. E., J. T. McLendon, L. W. Clements, Jr., and I. L. Feld. Characterization Studies of Florida

- Phosphate Slimes. BuMines RI 8089, 1975, 24 pp.
- 5. Jordan, C. E., C. W. Smith, G. V. Sullivan, and B. E. Davis. Recovery of Phosphate From Florida Phosphate Operations Slimes. BuMines RI 8731, 1982, 13 pp.
- 6. Trahar, W. J., and L. J. Warren. The Flotability of Very Fine Particles A Review. Int. J. Miner. Processing, v. 3, 1976, pp. 103-131.
- 7. Lawver, J. E., R. L. Wiegel, R. E. Snow, Sr., and C. L. Hwang. Phosphate Reserves Enhancement by Beneficiation. Min. Cong. J., Dec. 1982, pp. 27-31.